THE MANUFACTURE OF ARTIFICIAL GRAPHITE.

With the advance of electrochemistry it was found that electrodes of carbon were not so satisfactory as could be desired, because they disintegrated badly when employed in a great many of the electrochemical processes for which they were found to be practically the only substitute for the expensive platinum. Many attempts were therefore made to convert ordinary carbon into the more suitable modification—graphite—which possesses high conductivity and resistivity.

No very satisfactory method was devised, however, until Mr. Acheson succeeded in obtaining a very pure form accidentally when engaged in experiments upon the formation of silicon carbide, now known commercially as carborundum. Silicon carbide can be prepared by heating in the electric resistance furnace a mixture of silica and carbon, when the following reaction takes place:—

$$SiC_2+3C=SiC+2CO.$$

On one occasion, by the overheating of the furnace, he found that a large quantity of silicon carbide had been decomposed into carbon and silicon as follows:—

$$SiC=C+Si.$$

The silicon had actually been volatilised, and carbon in the form of graphite remained behind.

Silicon carbide is a magnificently crystalline product showing a splendid iridescence, and the graphite which was left behind had all the crystalline shape which the carbide originally had, but it no longer possessed iridescence, and in place of being next only in hardness to the diamond, was soft and friable, and had the familiar black appearance of graphite. Analysis showed it to be almost pure carbon, containing no more than 0·05 per cent. of impurities.

It might naturally be supposed that the purity of the final product would be directly affected by the purity of the starting material. This certainly would be so if it were simply a case of the high temperature of the furnace changing ordinary carbon into graphite; but as the reaction depends upon the formation of a carbide, which then at the great heat to which it is subjected loses one constituent by volatilisation, the other constituent remaining behind, and, furthermore, as at the temperature at which this reaction takes place all other metals are volatilised, or first converted into carbides and then volatilised, leaving the carbon, this is not so. In the preparation of graphite it is not necessary that sand in the proportions

$$SiO_2+3C=SiC+2CO$$

should be employed, because if the lower layers are converted into carbide volatilisation takes place. The vapourised silicon then passes through the next layer, and is converted into carbide; further volatilisation ensues, and the silicon passes through another layer, and so on progressively, until, finally, it passes out of the furnace as vapour.

The furnace is a long, oblong brick channel with electrodes at each end, which are connected together by means of a core made of granulated carbon. This core is surrounded by the mixture of sand and carbon, such as coke, from which the graphite is to be prepared. The furnace core is made of such dimensions that when the current is passed the temperature will be raised sufficiently high to convert the mixture surrounding it into carborundum, and then volatilise the silicon, leaving behind the graphite.

The carbon electrodes consist of twenty-five carbon rods, every rod being 86 cm. long and 10 cm. square. The internal part of the furnace is lined with silicon carbide, which protects the fire-bricks from the enormous operating heat. Generally, coal is employed as a raw material, being first broken to the size of peas; but as anthracite does not convey the current well, a conducting core of carbon rods is run through the centre of the furnace. When the furnace has been built up it is covered in with a mixture of sand and coke to prevent access of air. It is usually about 9 metres long, and the anthracite layer is 50 cm. by 35 cm. square. Such a furnace requires about 800 kilowatts to work it.

At the commencement of the operation the resistance is very high, therefore a high electrical pressure is required at the terminal electrodes. As the temperature rises and the core becomes graphitised, the resistance becomes less; consequently the voltage of the external circuit must be cut down. A pressure of about 200 volts is required at the commencement, but towards the end this is lowered to 75 volts. Shortly after the current is switched on, the volatile portions of the coal are driven off and burn with a characteristic yellow flame, which after a time becomes less, and its place is taken by the blue-coloured flame of carbon monoxide, because at this point the carbide commences to form. As the process continues the flame again becomes yellow, the carbide at this stage being decomposed. When the reduction is completed the flame is chiefly made up of burning volatilised metal, and shows a very fine absorption spectra. If a cold body is introduced into the flame it becomes covered with a felt-like coating of silicon dioxide.

The length of time required depends upon the purity of the graphite required. For most technical purposes it may contain up to 10 per cent. of ash. Further heating lowers the ash content, but, of course, as more energy is required, it increases the cost of the finished product.

Owing to the fact already mentioned, that much less than the theoretical amount of carbide-forming material need be added, Acheson finds that anthracite coal, which in its natural condition contains disseminated throughout its mass certain impurities, such as Fe_2O_3, SiO_2, Al_2O_3, &c., is particularly well adapted to produce graphite. That which contains 5·78 per cent. of impurities is especially suited for this purpose, and the graphite obtained from it contains only about 0·033 per cent. of ash. Other carbonaceous materials, such as brown coal, may be used, but, as a rule, the results are not so satisfactory.

It is not necessary that the carbide should be silicon carbide; other carbides, such as iron, appear to be of equal value in the formation of graphite. A spectrographic study of the flames produced in the operation always shows the presence of volatilised metals.

A particularly fine quality of graphite can be obtained from the coke left at the bottom of the stills used for cracking petroleum. This coke, known as petroleum coke, is honeycombed with small holes produced by the escaping gases, and the graphite obtained has exactly the same appearance as the original product, except that in place of the dull black of the coke it has the polished appearance characteristic of graphite.

For making electrodes, crucibles, motor brushes, and other articles of any particular shape, it is not necessary, as is the case with natural graphite, to grind up and then shape the articles with some suitable binding material; but by the Acheson process the articles are first made from some form of amorphous carbon, and afterwards converted directly into graphite. For example, lamp-black, powdered wood charcoal, or coke is mixed with a metal, its oxide or salt, which is capable of forming a carbide; the mixture is then moistened with water containing a little sugar—molasses or other binding material—made into a paste, and formed into any desired shape. It is then placed in the electric furnace, embedded in broken carbon, and covered over with amorphous carborundum to prevent loss of heat. The dimensions of the furnace are so arranged that when the current is passed the temperature is raised sufficiently high to graphitise a portion of the carbon; the furnace then becomes more conducting, and the pressure at the terminals can be lowered. As an example of the proportions of carbon and oxide used, the following has been found satisfactory for the formation of motor brushes:—ninety-seven parts finely powdered wood charcoal and three parts of iron oxide mixed into a paste and formed into the desired shape. If the electrodes are separated about 5 metres, the space between being filled with the articles to be graphitised and packed with carbon, an initial E.M.F. of 150 volts causes a current of 300 amperes. As the carbon and articles become graphitised the E.M.F. drops, until with a pressure of 100 volts a current of 7000 amperes is passing.

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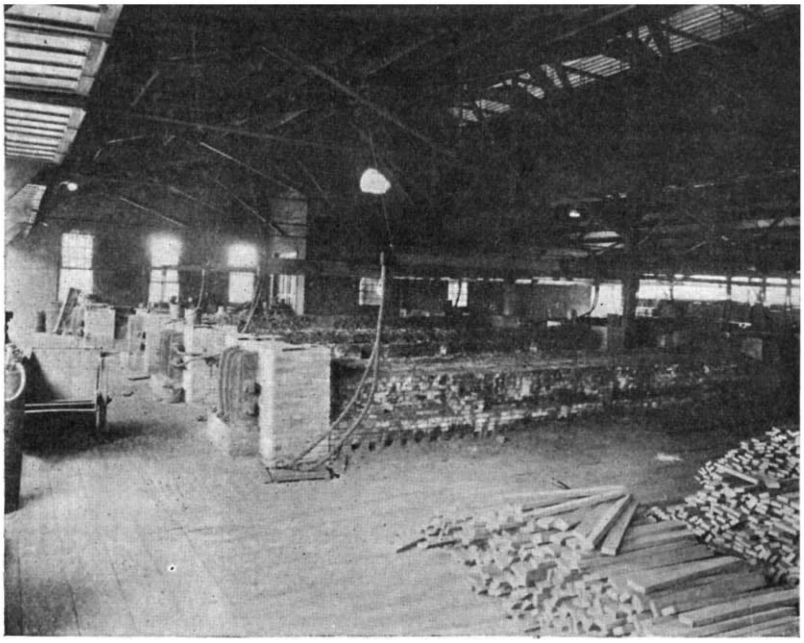
Acheson finds that it is not always desirable to convert the whole of the articles into graphite, but that if the operation is stopped when a portion is still ungraphitised they are stronger and less liable to fracture than when they consist of pure graphite.



International Acheson Graphite Co., Niagara Falls, N.Y., U.S.A.

It has already been mentioned that graphite of any desired purity can be obtained by this process, it simply being a question of how long the product is heated in the furnace. When it is to be ground and used as a lubricant it is necessary to make it of a higher degree of purity than when required for many other purposes; but, however pure the graphite, there are certain difficulties in employing it as a lubricant mixed with oil or water, owing to its precipitating out very shortly after being mixed with them. Although many attempts have been made to get over this difficulty, it is only quite recently that Acheson has been successful in doing so. In 1901, when experimenting upon the manufacture of crucibles, he found some difficulty in obtaining clay which had good binding qualities. He therefore commenced the study of clays which are used in the manufacture of crucibles. It was noticed that American crucible makers imported the clay from Germany, because, although the clay has practically the same chemical constitution as the American product, it has a greater tensile strength and is more plastic. Acheson then noticed that clays found at or near to the place at which the felspar rocks are decomposed are not so plastic or strong as when obtained at a distance from their source of origin. It occurred to him that this might be due to their containing vegetable or organic extract matter.

Experiments were therefore undertaken upon the action of vegetable extract matter, such as tannin and plant extracts, upon various clays. Remarkable results were obtained, it being found that a weak and only moderately plastic clay, after treatment with a dilute solution of tannic acid or extract of straw, increased in plasticity and became much stronger. In some cases the increased strength was as much as 300 per cent., and only 60 per cent. as much water was required to produce a given degree of fluidity. It remained suspended in water, and would pass through a filter paper. Clay thus treated Acheson called "Egyptianised," because the "Children of Israel" used straw in making bricks.

Now as clay so treated would remain suspended in water, it occurred to Acheson that perhaps the fine, unctuous graphite which he succeeded in directly manufacturing in the electric furnace in 1906 would also remain suspended in water if thus treated.

When disintegrated, graphite is treated with water containing tannin, the weight of which was from 3 per cent. to 6 per cent. of the graphite employed. The graphite remains suspended in the water indefinitely, and passes through a fine filter paper; it is therefore in a semi-colloidal condition. Graphite so treated Acheson calls "*deflocculated.*" To cause complete deflocculation and the suspension of the whole of the graphite requires prolonged mastication in the form of a paste with water and tannin, and after this mastication it is improved by diluting with considerable water and allowing to remain some weeks, with occasional stirring. The addition of a very small quantity of hydrochloric acid causes flocculation and precipitation.

The graphite, even after it has been flocculated, is in so fine a state of division that when dried by evaporation *en masse* it forms a hard cake. It is self-binding, like clay, and when dried in the sun is like a black clod of clay.

This deflocculated graphite is a splendid lubricator, and may be used in place of oil. It was tested on a shaft measuring 25/16 inches in diameter, and running at 3000 revolutions per minute in a bearing 10 inches long. On the same shaft a similar bearing was lubricated with oil, and this ran much the warmer of the two. If water alone is used for lubricating, rusting ensues; no rusting takes place with deflocculated graphite. Deflocculated graphite can also be suspended in the dehydrated state in oil. The two products come on the market as "aquadag" and "oildag" (d-a-g = deflocculated Acheson graphite).

Aquadag has been found very satisfactory as a cutting compound in screw-cutting. It will be readily understood that, while preventing rust, the high specific heat of the water permits high speed of the machinery, and therefore increased output. One disadvantage of aquadag is the rapid evaporation of the water; consequently, for general lubricating purposes, oildag is of more value. For automobile lubrication, for example, oildag is stated to have proved much more efficient than oil without graphite.

F. M. P.

THE FAUNA OF THE MAGELLAN REGION.[1]

IN 1892–3 Dr. W. Michaelsen conducted a zoological collecting expedition to the south end of South America, and was remarkably successful as regards booty. Descriptions of his collections began to appear in 1896, and they are now gathered together in three substantial volumes, each of several hundred pages. Dr. Michaelsen gives a lively account of his journeyings, and Prof. Dr. G. Pfeffer, of Hamburg, who persuaded some of his enlightened fellow-citizens to subsidise the expedition, refers briefly to the general bearing of the various contributions. It seems to us a matter for regret that there is no adequate summation of the results of the expedition, though we do not know what more Dr. Pfeffer could have done within the limits allotted to him. It is certainly

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[1] "Ergebnisse der Hamburger Magalhaensischen Sammelreise 1892–93. Herausgegeben vom Naturhistorischen Museum zu Hamburg. Bd. i., Allgemeines, Chordonier, Echinodermen, und Cœlenteraten. Bd. ii., Arthropoden. Bd. iii., Bryozoen und Würmer. Not continuously paged; numerous plates (Hamburg: L. Friederichsen and Co., 1896–1907.)

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